INTERNATIONAL STANDARD

ISO 3629

Second edition
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Photography — Processing chemicals — Specifications for potassium metabisulfite

Photographie — Produits chimiques de traitement — Spécifications relatives au métabisulfite de potassium

Reference number
ISO 3629:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3629 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 3629:1976), which has been technically revised.

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Introduction

0.1 This International Standard is one of a series that establishes criteria of purity for chemicals used in processing photographic materials. General test methods and procedures cited in this International Standard are compiled in parts 1, 5 and 9 of ISO 10349.

This International Standard is intended for use by individuals with a working knowledge of analytical techniques, which may not always be the case. Some of the procedures utilize caustic, toxic or otherwise hazardous chemicals. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles, rubber gloves and other protective apparel such as face masks or aprons where appropriate. Normal precautions required in the performance of any chemical procedure are to be exercised at all times but care has been taken to provide warnings for hazardous materials. Hazard warnings designated by a letter enclosed in angle brackets "⟨ ⟩" are used as a reminder in those steps detailing handling operations and are defined in ISO 10349-1. More detailed information regarding hazards, handling and use of these chemicals may be available from the manufacturer.

0.2 This International Standard provides chemical and physical requirements for the suitability of a photographic-grade chemical. The tests correlate with undesirable photographic effects. Purity requirements are set as low as possible consistent with these photographic effects. These criteria are considered the minimum requirements necessary to assure sufficient purity for use in photographic processing solutions, except that if the purity of a commonly available grade of chemical exceeds photographic processing requirements and if there is no economic penalty in its use, the purity requirements have been set to take advantage of the availability of the higher-quality material. Every effort has been made to keep the number of requirements to a minimum. Inert impurities are limited to amounts which will not unduly reduce the assay. All tests are performed on samples "as received" to reflect the condition of materials furnished for use. Although the ultimate criterion for suitability of such a chemical is its successful performance in an appropriate use test, the shorter, more economical test methods described in this International Standard are generally adequate.

Assay procedures have been included in all cases where a satisfactory method is available. An effective assay requirement serves not only as a safeguard of chemical purity but also as a valuable complement to the identity test. Identity tests have been included whenever a possibility exists that another chemical or mixture of chemicals could pass the other tests.

All requirements listed in clause 4 are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

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0.3 Efforts have been made to employ tests which are capable of being run in any normally equipped laboratory and, wherever possible, to avoid tests which require highly specialized equipment or techniques. Instrumental methods have been specified only as alternative methods or alone in those cases where no other satisfactory method is available.

Over the past few years, great improvements have been made in instrumentation for various analyses. Where such techniques have equivalent or greater precision, they may be used in place of the tests described in this International Standard. Correlation of such alternative procedures with the given method is the responsibility of the user. In case of disagreement in results, the method called for in the specification shall prevail. Where a requirement states "to pass test", however, alternative methods shall not be used.

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Photography — Processing chemicals — Specifications for potassium metabisulfite

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1 Scope

This International Standard establishes criteria for the purity of photographic-grade potassium metabisulfite and specifies the test methods to be used to determine the purity.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 10349-1:1992, *Photography — Photographic-grade chemicals — Test methods — Part 1: General.*

ISO 10349-5:1992, *Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content.*

ISO 10349-9:1992, *Photography — Photographic-grade chemicals — Test methods — Part 9: Reaction to ammoniacal silver nitrate.*

3 General

3.1 Physical properties

Potassium metabisulfite ($K_2S_2O_5$) exists in the form of white, glassy crystals. It has a relative molecular mass of 222,33.

3.2 Hazardous properties

Potassium metabisulfite is not hazardous when handled with normal precautions. Potassium metabisulfite is toxic if ingested. Avoid contact with acids.

3.3 Storage

Potassium metabisulfite shall be stored in a closed container at room temperature.

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4 Requirements

A summary of the requirements is shown in table 1.

5 Reagents and glassware

All reagents, materials and glassware shall conform to the requirements specified in ISO 10349-1 unless otherwise noted. The hazard warning symbols used as a reminder in those steps detailing handling operations are defined in ISO 10349-1. These symbols are used to provide information to the user and are not meant to provide conformance with hazardous labelling requirements as these vary from country to country.

6 Sampling

See ISO 10349-1.

7 Test methods

7.1 Assay

7.1.1 Specification

Content of potassium metabisulfite shall be 95,0 % (*m*/*m*) min.

7.1.2 Reagents

7.1.2.1 Acetic acid (CH_3COOH), approx. 2 mol/l solution.

Dilute 120 g of glacial acetic acid (DANGER: ⟨C⟩⟨S⟩ [1]) to 1 000 ml.

7.1.2.2 Neutral formaldehyde (HCHO), approx. 37 % (360 g/l) solution (DANGER: ⟨B⟩⟨C⟩⟨S⟩).

Adjust the pH of formaldehyde solution so that it is neutral to phenolphthalein indicator (7.1.2.8).

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Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	95,0 % (*m*/*m*) min.	7.1	ISO 3629
Heavy metals content (as Pb)	0,005 % (*m*/*m*) max.	7.2	ISO 10349-5
Iron content (Fe)	0,005 % (*m*/*m*) max.	7.3	ISO 10349-5
Reaction to ammoniacal silver nitrate	To pass test	7.4	ISO 10349-9
pH value	3,7 to 4,6	7.5	ISO 3629
Thiosulfate content (as $S_2O_3^{2-}$)	0,04 % (*m*/*m*) max.	7.6	ISO 3629
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.7	ISO 3629
NOTE — *m*/*m* = mass/mass			

1) Hazard warning codes are defined in ISO 10349-1.

7.1.2.3 Iodine (I_2), standard volumetric solution of 0,05 mol/l (12,7 g/l) [2) 3)].

Weigh, to the nearest 0,001 g, 12,7 g of freshly sublimed iodine (DANGER: ⟨C⟩⟨O⟩) into a tared weighing flask. Add 36 g of potassium iodide (KI) and 100 ml of water. After solution is complete, add three drops of hydrochloric acid (7.1.2.9) (⟨C⟩⟨B⟩), and dilute to 1 litre at 20 °C in a volumetric flask. From the mass of iodine, m, calculate the concentration, c, in moles per litre, from

$$c = m/254$$

7.1.2.4 Salicylic acid ($HOC_6H_4CO_2H$), 1 % (10 g/l) solution.

7.1.2.5 Starch indicator, 5 g/l.

Stir 5 g of soluble starch into 100 ml of salicylic acid (7.1.2.4). Add 300 ml to 400 ml of boiling water and boil until the starch dissolves. Finally, dilute to 1 000 ml with water.

7.1.2.6 Sodium thiosulfate ($Na_2S_2O_3$), standard volumetric solution of 0,100 mol/l (15,8 g/l) [2)].

7.1.2.7 Sulfuric acid (H_2SO_4), 0,05 mol/l (4,9 g/l) solution [2) 4)].

7.1.2.8 Phenolphthalein indicator, 5 g/l.

Dissolve 1 g of phenolphthalein in 100 ml of 95 % (V/V) ethanol and add 100 ml of water with constant stirring. Filter if necessary.

7.1.2.9 Hydrochloric acid (HCl), $\rho \approx 1{,}18$ g/ml (DANGER: ⟨C⟩⟨B⟩).

7.1.3 Apparatus

7.1.3.1 Burette, of 50 ml capacity.

7.1.3.2 Pipette, of 50 ml capacity.

7.1.4 Procedure

Using a pipette (7.1.3.2), deliver 50,00 ml of the iodine solution (7.1.2.3) into a flask. Weigh, to the nearest 0,000 1 g, a test portion of about 0,23 g and wash this into the flask. Add 5 ml of the acetic acid (7.1.2.1) and mix to ensure complete dissolution of the sample. Titrate with the sodium thiosulfate (7.1.2.6), adding the starch indicator (7.1.2.5) just before the endpoint.

Weigh, to the nearest 0,001 g, another test portion of about 5 g. Dissolve it in 50 ml of water and add 50 ml of the formaldehyde (7.1.2.2). Add a few drops of the phenolphthalein indicator (7.1.2.8) and titrate with the sulfuric acid (7.1.2.7) to the colour change.

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2) Commercially available analysed reagent solutions are recommended. If solutions are to be prepared, see any quantitative analytical chemistry text.

3) It is recommended that self-prepared iodine solutions be standardized before use.

4) This can be prepared from concentrated sulfuric acid, $\rho \approx 1{,}84$ g/ml (DANGER: ⟨⟨C⟩⟩).

7.1.5 Expression of results

The assay, expressed as a percentage by mass, for potassium metabisulfite ($K_2S_2O_5$), is given by

$$\frac{5{,}558(100 \cdot c_1 - c_2 \cdot V_2)}{m_1} - \frac{11{,}12 \cdot c_3 \cdot V_3}{m_2}$$

where

c_1 is the actual concentration, expressed in moles per litre, of the iodine solution (7.1.2.3);

c_2 is the actual concentration, expressed in moles per litre, of the sodium thiosulfate (7.1.2.6);

c_3 is the actual concentration, expressed in moles per litre, of the sulfuric acid (7.1.2.7);

V_2 is the volume, in millilitres, of the sodium thiosulfate (7.1.2.6) used for titration;

V_3 is the volume, in millilitres, of the sulfuric acid (7.1.2.7) used for titration;

m_1 is the mass, in grams, of the test portion used for the first titration;

m_2 is the mass, in grams, of the test portion used for the second titration;

100 is twice the volume, in millilitres, of I_2 added (7.1.4);

5,558 is a conversion factor obtained from the mass of potassium metabisulfite equivalent to 1 mole of iodine (i.e. 55,58 as there are 4 equivalents of sodium metabisulfite per mole of iodine) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage);

11,12 is a conversion factor obtained from the mass of potassium metabisulfite equivalent to 1 mole of potassium sulfite present (i.e. 111,2 as there are 2 equivalents of sodium sulfite per mole of sodium metabisulfite) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

NOTE — When an assay based on sulfite content but expressed as potassium metabisulfite ($K_2S_2O_5$) is desired, the second titration in 7.1.4 is not required and the assay is given by

$$\frac{5{,}558(100 \cdot c_1 - c_2 \cdot V_2)}{m_1}$$

7.2 Heavy metals content

7.2.1 Specification

Maximum content of heavy metals shall be 0,005 % (*m/m*).

7.2.2 Procedure

NOTE — The standard for the iron test (7.3) is prepared in the same way as the heavy metals standard.

Determine the percentage of heavy metals in accordance with ISO 10349-5. Use a test portion of 1,90 g to 2,10 g, prepared in accordance with ISO 10349-5:1992, 7.3. Use 10 ml of the heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

7.3 Iron content

7.3.1 Specification

Maximum content of iron shall be 0,005 % (*m/m*).

7.3.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of 1,90 g to 2,10 g of the sample, prepared in accordance with ISO 10349-5:1992, 7.3. Use 10 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

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7.4 Reaction to ammoniacal silver nitrate

7.4.1 Specification

To pass test.

7.4.2 Procedure

Determine the reaction to ammoniacal silver nitrate in accordance with ISO 10349-9.

7.5 pH value

7.5.1 Specification

The pH shall be between 3,7 and 4,6.

7.5.2 Apparatus

7.5.2.1 Electronic pH-meter, equipped with a glass electrode and standard reference electrode.

7.5.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g. Dissolve it in about 80 ml of boiled and cooled water and dilute to 100 ml. Measure the pH of the solution at 20 °C, using the pH-meter (7.5.2.1) in accordance with the manufacturer's instructions.

7.6 Thiosulphate content (as $S_2O_3^{2-}$)

7.6.1 Specification

The maximum thiosulfate content as $S_2O_3^{2-}$ shall be 0,04 % (*m/m*).

7.6.2 Reagents

7.6.2.1 Mercury(II) chloride reagent

Dissolve 25 g of potassium bromide (KBr) and 25 g of mercury(II) chloride ($HgCl_2$) (DANGER: ⟨⟨S⟩⟩) in 900 ml of water at 50 °C. Cool, dilute to 1 000 ml and allow to stand overnight. Filter if not perfectly clear.

7.6.2.2 Thiosulfate standard, 1 ml contains 0,056 mg $S_2O_3^{2-}$.

Dilute 5 ml of sodium thiosulfate solution (7.1.2.6) to 1 000 ml.

7.6.3 Apparatus

7.6.3.1 Graduated pipette, of 1 ml capacity.

7.6.3.2 Two matched Nessler colour-comparison cylinders, of 50 ml capacity.

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